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नमूने लेने और परीक्षण की पद्धतियाँ प्राकृतिक और संश्लेषित सुगन्ध सामग्री

भाग 7 अम्ल का मान ज्ञात करना

(तीसरा पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 7 DETERMINATION OF ACID VALUE

(Third Revision)

ICS 71.100.60

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 2

Natural and Synthetic Fragrance Materials Sectional Committee, PCD 18

NATIONAL FOREWORD

This Indian Standard (Part 7) (Third Revision) which is identical with ISO 1242 : 1999 'Essential oils — Determination of acid value' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Natural and Synthetic Fragrance Materials Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of the ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminology and conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

The technical committee responsible for the preparation of this standard has reviewed the provisions of ISO 356 'Essential oils — Preparation of test samples' and decided that it is acceptable for use in conjunction with this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 7 DETERMINATION OF ACID VALUE

(Third Revision)

1 Scope

This International Standard specifies a method of determining the acid value of essential oils. This method is not applicable to essential oils containing appreciable quantities of lactones.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 356, *Essential oils — Preparation of test sample.*

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 385-2, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

ISO 385-3, *Laboratory glassware — Burettes — Part 3: Burettes for which a waiting time of 30 s is specified.*

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
acid value (AV)
number of milligrams of potassium hydroxide required to neutralize the free acids contained in 1 g of the essential oil

4 Principle

The free acids are neutralized with a standardized ethanolic solution of potassium hydroxide.

5 Reagents

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

5.1 Ethanol, 95 % (by volume) at 20 °C, freshly neutralized with the potassium hydroxide solution (5.2), in the presence of the coloured indicator (5.3) used for the determination

5.2 Potassium hydroxide, standard solution in ethanol, $c(\mathrm{KOH}) = 0{,}1$ mol/l, standardized before each set of tests.

5.3 Coloured indicator

Use either:

a) phenolphthalein, 2 g/l solution in ethanol (5.1), or

b) phenol red, 0,4 g/l solution in ethanol [20 % (by volume)] if the essential oil contains phenolic groups.

NOTE The corresponding monographs will specify which is to be used.

6 Apparatus

Ordinary laboratory apparatus and, in particular, the following.

6.1 Flask, of capacity 100 ml.

NOTE If subsequent determination of the ester value is desired, the saponification device is indicated in ISO 709.

6.2 Measuring cylinder, of capacity 5 ml.

6.3 Burette, of capacity 2 ml, graduated in 0,01 ml intervals, conforming to the requirements for class A given in the relative part of ISO 385.

6.4 Analytical balance, of precision 0,001 g.

7 Sampling

It is important that the laboratory receive a representative sample, not damaged or modified during transport or storage before arrival at the laboratory.

The sampling method is not included in this International Standard. A recommended sampling method is given in ISO 212.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 356.

9 Procedure

9.1 Test portion

Weigh, to the nearest 0,5 mg, approximately 2 g of the test sample.

NOTE If the size of the test portion is to be different from the amount stated above, this will be specified in the standards specific to the essential oil concerned.

9.2 Determination

Introduce the test portion (9.1) into the flask (6.1). Add 5 ml of neutralized ethanol (5.1) and no more than 5 drops of the indicator (5.3), either phenolphthalein solution or phenol red solution depending on the case. Titrate the liquid with the potassium hydroxide solution (5.2) contained in the burette (6.3).

Continue the addition until a change in colour that persists for 30 s, is achieved. Note the volume (V) of potassium hydroxide used.

NOTE If it is required to determine the ester value, reserve the flask and its contents. This content is designated as (A) in ISO 709.

10 Calculation

The acid value (AV) is given by the formula:

$$\mathrm{AV} = V \cdot c \cdot \frac{56{,}11}{m}$$

where

V is the volume, in millilitres, of potassium hydroxide solution (5.2) used in the titration;

c is the exact concentration, in moles per litre, of the potassium hydroxide solution;

m is the mass, in grams, of the test portion.

If the volume of potassium hydroxide used is less than 0,1 ml, repeat the test on a larger test portion.

Express the result to one decimal place.

11 Precision

11.1 Repeatability

The difference between two independent single test results, obtained by means of this method with the same essential oil tested in the same laboratory and by the same operator using the same apparatus in a short period of time, will in not more than 5 % of cases be greater than 0,05 in absolute terms or greater than 2,5 % in relative terms, taking into account the greatest value for a given result.

11.2 Reproducibility

The difference between two individual test results, obtained by means of this method with the same essential oil tested in different laboratories by different operators using different apparatus, will in not more than 5 % of cases be greater than 0,1 in absolute terms or greater than 5 % in relative terms, taking into account the greatest value for a given result.

12 Test report

The test report shall include:

- the test method used, with reference to this International Standard;
- the test result(s) obtained; and
- if the repeatability has been verified, the final result obtained.

It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that might have influenced the results.

The test report shall include all details required for the complete identification of the sample.

Bibliography

[1] ISO 212, *Essential oils — Sampling.*

[2] ISO 709, *Essential oils — Determination of ester value.*

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Amendments Issued Since Publication

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